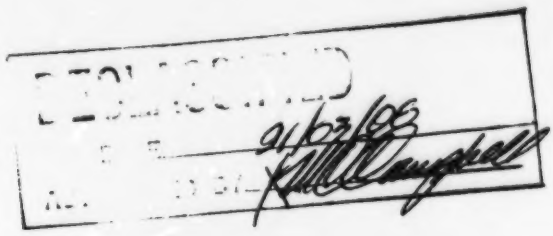

PROGRESS REPORT ON RECOVERY OF RARE EARTHS BY SOLVENT EXTRACTION

by

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ABSTRACT

Preliminary studies on the extraction of rare earths from a sulphuric acid leach liquor of a mineral sample from Raretec Inc have indicated that a thorium extraction step is needed before the rare earths can be extracted. Primene JMT or Adogen 283 are effective in extracting the thorium.

OBJECTIVE

The objective of this solvent extraction program was to extract and separate rare earth elements from the sulfuric acid leach liquor of the ore sample supplied by Raretec Minerals Inc.

TEST PROCEDURE

Sulfuric acid leaching solution was provided by Dr. Haque of the Leach Section. To maximize the amount of feed solution for the solvent extraction tests, 24 hours leach pregnant, repulp and water wash liquor were mixed. Filtration of the feed was necessary because of the occurrence of white precipitation after combining those three solutions. Silicon was the principal constituent of the precipitate as identified by semi-quantitative spectrochemical analysis.

Successive shake-out bench tests were conducted in separatory funnels. A mixture of 10% PA216 (phosphonic acid from Hoechst) and 5% (tri-n-butyl phosphate) in Varsol 140 was chosen, because of its good extraction ability for rare earths and its rejection of iron, based on the results of earlier screening tests carried out at CANMET. Fresh organic was added to contact the raffinate remaining after each shake-out. Contact times were always two minutes at A/O 10/1, with the exception of the first contact at A/O 5/1. Stripping followed each extraction at O/A 1/1 for two minutes with 50% HCl.

RESULTS AND DISCUSSION

The components of the solvent extraction feed and the results of the tests are shown in Table 1. Poor extraction of the total rare earth content was observed. Fractionation, that is the separation of the rare earth elements with the successive extractions, was not observed in these tests.

Table 1. Extraction with 10% PA216 + 5% TBP for rare earths

No. of contact	1 contact A/O = 5			2 contact A/O = 10			3 contact A/O = 10			4 contact A/O = 10			
Elements	Feed (g/L)	Raff. (g/L)	% Extracted	Feed (g/L)	Raff. (g/L)	% Extracted	Feed (g/L)	Raff. (g/L)	% Extracted	Feed (g/L)	Raff. (g/L)	% Extracted	Total Extracte
Y	.30	.25	17	.24	0.19	21	0.19	0.16	16	0.16	0.15	6	50
La	4.26	4.28	~0	4.16	4.20	~0	4.20	4.13	~0	4.13	4.17	~0	~0
Ce	6.64	6.69	~0	6.52	6.56	~0	6.56	6.47	~0	6.47	6.53	~0	~0
Pr	0.57	0.58	~0	0.57	0.57	~0	0.57	0.56	2	0.56	0.56	~0	~2
Nd	1.80	1.81	~0	1.79	1.77	~1	1.77	1.75	~1	1.75	1.75	0	~3
Sm	0.26	0.26	0	0.26	0.26	0	0.26	0.25	~0	0.25	0.25	0	~0
Eu	0.048	0.047	2	0.047	0.046	2	0.046	0.046	0	0.046	0.046	0	~0
Gd	0.18	0.18	0	0.18	0.18	0	0.18	0.17	~0	0.17	0.17	0	~0
Tb	<.022	<.02	~0	<.022	<.02	~0	<.022	<.02	~0	<.022	<.02	~0	~0
Dy	0.13	0.12	~10	0.11	0.11	0	0.11	0.10	~10	0.10	0.10	0	~20
Ho	<.023	<.02	~0	<.022	<.02	~0	<.022	<.02	~0	<.021	<.02	~0	~0
Er	.053	0.40	25	.040	.020	50	.020	<.020	~0	<.020	<.020	~0	>62
Yb	<.031	<.020	~35	<.026	<.020	~20	<.023	<.020	~0	<.022	<.020	~0	>36
Lu	<.022	<.020	~10	<.021	<.020	~5	<.020	<.020	~0	<.020	<.020	~0	~10
U	.017	.0047	72	.0044	.0029	34	.0029	.0016	45	.0016	.0017	~0	90
Th	1.18	0.15	87	0.15	0.055	63	0.055	0.02	64	0.02	0.01	50	99

This low extractability may be due to an unexpectedly high thorium level in the feed solution. The results show that thorium, which is one of the major constituents of the leach solution, is extracted preferentially to the rare earth elements.

THORIUM EXTRACTION

Several bench tests were conducted to extract thorium prior to extracting rare earths. The primary amine, Primene JMT (Rohm & Haas), and secondary amines, Amberlite LA-1, LA-2 (Rohm & Haas) and Adogen 283 (Sherex) were tried at 0.1M strength in Varsol 140 using A/O 1/1 with two minutes contact time. In Table 2 are shown the screening extraction results and separation factors for Th/Fe using four solvents. Primene JMT gives 90% extraction with 0.8 separation factor; Adogen 283 gives 88% extraction with 1.27 separation factor. Although the equilibrium isotherm and kinetic tests were done with Primene JMT, in general Adogen 283 was found to be the best extractant for thorium. The Amberlite LA-1 and LA-2 were not as effective.

The physical performance of the thorium extraction was poor in all cases, the worst being the Amberlites. The leach feed liquor was not stable and tended to precipitate after contact with the organic. This is a direct reflection of some of the problems observed in the leaching and the situation should improve as the leach is optimized. No work was carried out to improve separation times, for example by adding modifiers or changing diluents.

The results shown in Table 3 of the equilibrium isotherm data indicate that with 0.1M Primene JMT only 1 or 2 stages are required to reduce thorium to less than 0.01 g/L in the raffinate. Very little rare earths were co-extracted, except with A/O ratio higher than 5/1. Iron was co-extracted with a maximum amount of 1.69 g/L. Based on the results described above, it is suggested that successive contact with fresh feed be examined to determine whether thorium is extracted preferentially to iron using Primene JMT or Adogen 283. Scrubbing may be needed to recover pure thorium product.

Kinetic tests showed that the equilibrium was reached in 50 seconds, and that the co-extraction of iron was not affected by varying the contact time.

Table 2. Screening for thorium extractants
A/O 1/1, Feed: Th 1.08, U .088, Fe 11.62 g/L

Reagents	Th			Fe			U		Separation time (min)	Remarks
	Raff. (g/L)	Solvents (g/L)	% Extraction	Raff. (g/L)	Solvent (g/L)	SF Th/Fe	Raff. (g/L)	Solvent (g/L)		
LA-1	.27	.71	75.0	11.5	.75	40.3	<.018	.073	60	Aq. crud, org. yellow, clear
LA-2	.78	.32	27.8	9.67	1.67	2.4	<.018	.073	70	Aq. milky, org. clear
Adogen 283	.12	1.03	88.8	12.8	.81	136	<.018	.076	50	Aq. very little crud
Primene JMT	.10	1.05	90.7	11.72	1.27	97	<.05	<.011	>30	Aq. milky

Table 3. Extraction isotherm with 0.1M Primene JMT

A/O	Th			Fe			U		
	Raff. (g/L)	Solvents (g/L)	% Extraction	Raff. (g/L)	Solvent (g/L)	SF Th/Fe	Raff. (g/L)	Solvent (g/L)	
1/10	<.019	.1	100	4.02	.24	> 88	.2	.017	Feed components
1/5	<.019	.24	100	8.49	.53	> 202	<.02	<.011	Th 1.08 g/L
1/1	.1	1.05	90.74	11.72	1.27	97	<.02	<.011	U .088 g/L
6/1	.47	2.89	56.48	9.14	.013	4320	<.02	<.011	Fe 11.62 g/L

CONCLUSION

It can be concluded that little success can be achieved in the direct extraction and fractionation of rare earths from Raretec ore sulfuric acid leach liquor using 10% PA216 + 5% TBP. This is primarily due to preferential extraction of thorium. To overcome this, an additional thorium extraction circuit is needed prior to rare earths extraction. The experimental results show that primary amines, such as Primene JMT and secondary amines, such as Adogen 283 are effective in the extraction of thorium. Stripping and scrubbing tests are required to complete the circuit for removing thorium.

Countercurrent successive shake-out bench tests should be conducted using leach liquor with thorium being removed. This may enhance the fractionation of the rare earths group. Additional testwork will be required to establish the design parameters of the rare earth circuit. Further tests, both for the thorium and rare earth circuits will, however, be dependent upon the availability of sufficient quantities of leach liquor.